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IS 10614 (1983): Method for atomic absorption spectrophotometric determination of sodium and potassium [CHD 1: Inorganic Chemicals]



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IS : 10614 - 1983

*Indian Standard*

METHOD FOR ATOMIC  
ABSORPTION SPECTROPHOTOMETRIC  
DETERMINATION OF SODIUM AND  
POTASSIUM

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**INDIAN STANDARDS INSTITUTION**  
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG  
NEW DELHI 110002

# Indian Standard

## METHOD FOR ATOMIC ABSORPTION SPECTROPHOTOMETRIC DETERMINATION OF SODIUM AND POTASSIUM

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## *Indian Standard*

# METHOD FOR ATOMIC ABSORPTION SPECTROPHOTOMETRIC DETERMINATION OF SODIUM AND POTASSIUM

### 0. FOREWORD

**0.1** This Indian Standard was adopted by the Indian Standards Institution on 15 June 1983, after the draft finalized by the Chemical Standards Sectional Committee had been approved by the Chemical Division Council.

**0.2** The atomic absorption spectrophotometric method is based on the fact that all elements when atomised will absorb light of the same wavelength, that they emit when excited. When a solution containing salts of sodium and potassium is introduced in the flame, the intensity of the transmitted radiation will decrease in proportion to the amount of ground state atoms present in the flame, that is, extent of the concentration of these elements in the solution. A hollow cathode lamp, made of the element to be determined, provides the source of radiation. The metal atoms to be measured are fed to the flame by aspirating the solution into the flame. A monochromator eliminates the unwanted radiation from the flame and permits mainly the radiation from the hollow cathode lamp to fall on the photosensitive device. The difference between the intensity of absorbed and reference beam is taken as the measure of concentration of the analysed element.

**0.3** In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960\*.

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### 1. SCOPE

**1.1** This standard prescribes the method for determination of sodium and potassium, employing atomic absorption spectrophotometric technique.

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\*Rules for rounding off numerical values ( *revised* ).

## **2. QUALITY OF REAGENTS**

**2.1** Unless specified otherwise, pure chemicals and deionized water shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

## **3. OUTLINE OF THE METHOD**

**3.1** The material is powdered and homogenized, so as to pass through 150 micrometre IS sieve. A known mass of the air-dried sample is suitably dissolved in acid or acid mixtures, filtered and diluted to definite volume. This solution or its suitable dilutions are made to aspirate into the flame. Standards are also aspirated in the same way. If elements are to be determined in metals, they may be brought into solution using suitable acids.

## **4. INTERFERENCE**

**4.1** No chemical interferences have been found. Sodium and potassium are partially ionized in air-acetylene flame (to suppress ionization add potassium nitrate or potassium chloride to give final concentration of 2 000  $\mu\text{g/ml}$  of potassium in the solution, in which sodium has to be measured and add cesium nitrate or cesium chloride to give a final concentration of 1 000  $\mu\text{g/ml}$  cesium in all solution including blank where potassium has to be measured). By appreciable concentration of mineral acids present in the solution, the absorbance reading is reduced for both sodium and potassium. Hydrochloric, sulphuric, nitric and phosphoric acids cause decrease in the reading. Influence of nitric and sulphuric acid is mainly due to changes in physical properties and the aspiration rates of the solution. The influence of hydrochloric acid and phosphoric acid is due to the evaporation process in the flame.

**4.2** Up to 160 ppm of aluminium, phosphate, sulphate and silicate do not interfere with low concentration of sodium, namely 2-10 ppm. But it is found that slight decrease in absorption occurs in the presence of high concentration of phosphates. But the same amount of aluminium, phosphate, sulphate and silicate do not interfere in potassium determination in the presence of aluminium chloride (1 M).

## **5. APPARATUS**

**5.1 Atomic Absorption Spectrophotometer** — It shall have following parameters.

**5.1.1 Lamp Current** — As recommended by the instrument maker (For instance, Perkin Elmer atomic absorption spectrophotometer suggests 8 m A and 12 m A for Na and K respectively).



**5.1.2 Fuel** — acetylene.

**5.1.3 Support** — air.

**5.1.4 Flame Stoichiometry** — oxidizing air-acetylene flame.

**5.1.5 Wavelength and Working Range** — It shall be as follows:

Wavelength, nm		Band Pass*, nm		Working Range, µg/ml	
Sodium	Potassium	Sodium	Potassium	Sodium	Potassium
589.0	766.5	0.2	0.5	0.15 to 0.60	0.5 to 2.0
589.6	769.9	0.2	0.5	0.5 to 2.0	1.5 to 6.0
330.2 } 330.3 }	404.4	0.2	0.1	100 to 400	200 to 800

NOTE — By rotation of burner on 589 and 589.6 nm for sodium and on 766.5 and 769.9 nm for potassium, measurements can also be taken for 20 to 50 µg/ml.

\*This can be changed as per the instrument specification.

## 6. REAGENTS

**6.1 Concentrated Hydrochloric Acid** — see IS : 265-1976\*.

**6.2 Concentrated Nitric Acid** — see IS : 264-1976†.

**6.3 Hydrofluoric Acid**

**6.4 Sodium Chloride**

**6.5 Potassium Chloride**

**6.6 Cesium Chloride**

## 7. PROCEDURE

**7.1 Standard Sodium Solution** — Weigh exactly 2.542 g of sodium chloride, previously dried at 120°C. Transfer the salt with water to 250-ml beaker. Dissolve in 100 ml of water and add 10 ml of hydrochloric acid. Transfer the solution to 1-litre volumetric flask and make up the volume, mix well and store in polyethylene bottle. This solution contains 1 mg/ml of sodium. For preparing standard solution, dilute the above stock solution taking appropriate aliquot. Add 0.4 g of potassium chloride per 100 ml to have 2 000 ppm of potassium to suppress ionization while working in air-acetylene flame.

\*Specification for hydrochloric acid ( second revision ).

†Specification for nitric acid ( second revision ).

**7.2 Standard Potassium Solution** — Weigh exactly 1.9068 g of potassium chloride dried at 400°C and cooled to room temperature. Transfer to 250 ml beaker, dissolve in 100 ml of water and add 10 ml of hydrochloric acid. Transfer to 1-litre volumetric flask and make up to the mark and store in polyethylene bottle. This solution contains 1 mg/ml of potassium. Take aliquot of this solution and accordingly add about 0.13 g of cesium chloride per 100 ml volume, make up solution to have about 1 000 ppm of cesium in the working solution, if the measurements are taken in air-acetylene flame. In air-propane flame only further dilution are required without cesium chloride addition.

NOTE — Sodium chloride/sodium nitrate may also be used for cesium chloride.

### 7.3 Preparation of Sample Solution

**7.3.0** There can be numerous matrices in which sodium and potassium may be present and to bring these metals in solution, the method may vary depending on the nature of matrix. Some of the common matrices wherein sodium and potassium are generally determined have been included in this standard.

**7.3.1 Minerals** — Powder the material so that it passes through 150 micrometer IS sieve. Weigh accurately a suitable mass of the material (dried at 110°C and cooled) in a 100-ml beaker. Add 10 ml of hydrochloric acid, 5 ml of nitric acid and 10 ml of water. Boil till the reaction is completed. Evaporate to syrupy consistency. Extract the residue with hydrochloric acid (2 percent) and filter if necessary. Transfer the filtrate to 100-ml volumetric flask. Treat the residue, if any, on the filter paper as given in 7.3.2.

**7.3.2 Silicate** — Decompose the silicate (0.2 g) with 5 ml hydrofluoric acid and 10 ml of concentrated nitric acid in a platinum dish or Teflon beaker and evaporate just to dryness or syrupy consistency. Repeat this process, if necessary, for complete decomposition. Add 10 ml of concentrated nitric acid and evaporate again. Transfer to 100 ml beaker and add 10 ml of concentrated hydrochloric acid and evaporate again to syrupy consistency. Take in hydrochloric acid (1 percent) solution, boil and filter to make up 100 ml.

**7.3.3 Organics** — Incinerate the sample at 400 to 450°C till the carbon decomposes, then proceed as given in 7.3.2.

**7.3.4 Effluents and Sewages** — Take 100 ml of sample in a beaker and add 5 ml of concentrated nitric acid and evaporate to dryness. Repeat this operation once more adding concentrated nitric acid. Dissolve the residue in minimum volume of concentrated hydrochloric acid. Boil to dissolve, dilute to 50 ml with water, filter and make up to 100 ml, if necessary, potassium chloride or cesium chloride may be added accordingly.

**7.3.5 Water** — If the water is clear, just acidify with hydrochloric acid and feed directly into the flame ( if necessary cesium chloride or potassium chloride may be added to suppress ionization of potassium and sodium respectively in air-acetylene flame ) otherwise filter and proceed in the same manner.

**7.4 Determination** — Aspirate the appropriate solution to the flame along with the required standard solution and note the absorption. Take at least four standards in the range in which sample absorption comes. Plot the graph and find out the exact concentration of the elements in the solution.

NOTE — Whenever, a direct reading instrument ( concentration in  $\mu\text{g/ml}$  ) is used, a blank containing the matrix, along with blank containing known standard of the element to be determined is also aspirated and after standardizations, the test solution may be aspirated and concentration directly read out in  $\mu\text{g/ml}$ .

### 8. CALCULATION

$$\text{8.1 Sodium or potassium, percent by mass} = \frac{e \times V}{10^6} \times \frac{100}{M}$$

where

$e$  = concentration of sodium or potassium in  $\mu\text{g/ml}$  in the final solution,

$V$  = volume in ml of the final solution, and

$M$  = mass in g of the material in the final solution.

# INTERNATIONAL SYSTEM OF UNITS ( SI UNITS)

## Base Units

QUANTITY	UNIT	SYMBOL
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

## Supplementary Units

QUANTITY	UNIT	SYMBOL
Plane angle	radian	rad
Solid angle	steradian	sr

## Derived Units

QUANTITY	UNIT	SYMBOL	DEFINITION
Force	newton	N	$1 \text{ N} = 1 \text{ kg.m/s}^2$
Energy	joule	J	$1 \text{ J} = 1 \text{ N.m}$
Power	watt	W	$1 \text{ W} = 1 \text{ J/s}$
Flux	weber	Wb	$1 \text{ Wb} = 1 \text{ V.s}$
Flux density	tesla	T	$1 \text{ T} = 1 \text{ Wb/m}^2$
Frequency	hertz	Hz	$1 \text{ Hz} = 1 \text{ c/s (s}^{-1}\text{)}$
Electric conductance	siemens	S	$1 \text{ S} = 1 \text{ A/V}$
Electromotive force	volt	V	$1 \text{ V} = 1 \text{ W/A}$
Pressure, stress	pascal	Pa	$1 \text{ Pa} = 1 \text{ N/m}^2$